

Data Validation Checklist Semivolatile Organic Analyses

Project: 35TH Avenue Superfund Site
 Laboratory: TestAmerica - Savannah, GA
 Method: SW-846 8082A (PCBs)
 Matrix: Soil
 Reviewer: Nicole Lancaster
 Concurrence¹: Martha Meyers-Lee

Project No: 15268508.20000
 Job ID.: 680-85785-5
 Associated Samples: CV0705C-GS (680-85785-24)
 Samples Collected: 12/12/2012
 Date: 02/18/2013
 Date: 02/26/2013

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
1. Were sample storage and preservation requirements met? If temperature >6°C, then J/UJ-flag results.	✓				
2. Were all COC records signed and integrity seals intact, indicating that COC was maintained for all samples?	✓				
3. Were there any problems noted in laboratory data package concerning condition of samples upon receipt?		✓			
4. Do any soil samples contain more than 50% water? If yes, then results are to be reported on a wet-weight basis.		✓			
5. Were holding times met (≤7 and 14 days from collection to extraction for aqueous and solid samples, respectively; ≤40 days from extraction to analysis)? If not, then J/UJ-flag sample results. If grossly (2x) exceeded, then flag J/R.	✓				
6. Were results for all project-specified target analytes reported?	✓				
7. Were project-specified Reporting Limits achieved for undiluted sample analyses?	✓				
8. Were samples with analyte concentrations exceeding the calibration range of the instrument re-analyzed at a higher dilution? If not, then J-flag sample result.	✓				
9. Was a method blank extracted with each batch (i.e., one per 20 samples, per batch, per matrix and per level)?	✓				
10. Were target analytes detected in the method blank?		✓			
11. Were target analytes detected in equipment/rinsate blanks?			✓	Rinsate blank 121112-RB-Shovel (680-85731-47) was not analyzed for PCBs.	
12. Are equipment/rinsate blanks associated with every sample? If		✓		According to the QAPP, a rinsate blank is to be	

¹ Independent technical reviewer
 URS Group, Inc.
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Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
no, note in DV report.				collected after each decontamination event, which occurs once per week per the client. The rinsate blank, 121112-RB-Shovel (680-85731-47), collected during the week of December 10, 2012 was analyzed for only PAHs and metals.	
13. Were analytes detected in samples below the blank contamination action level? If yes, U-flag positive sample results <5x associated blank concentration (10x for common blank contaminants – phthalates)			✓	Blank contamination does not exist.	
14. Is a field duplicate associated with this Job?		✓			
15. Was precision deemed acceptable as defined by the project plans?			✓		
16. Were DFTPP ion abundance criteria (i.e., Table 3 of SW-846 8270C) met? If no, professional judgment may be applied to determine to what extent the data may be utilized.			✓		
17. Were samples analyzed within 12 hours of the DFTPP tune? If no, professional judgment may be applied to determine to what extent the data may be utilized.			✓		
18. Were initial and continuing calibration standards analyzed at the proper frequency for each instrument? <ul style="list-style-type: none"> Ensure that a minimum of five standards are used for the initial calibration. If no, use professional judgment to determine the effect on the data and note in the reviewer narrative. An initial calibration is to be associated with each sample analysis. A continuing calibration standard is to be analyzed for every 12 hours of sample analysis per instrument. 	✓			8082A <ul style="list-style-type: none"> Instrument ID: SGZ Initial Calibration: 12/18/12 (6-point curve for 1016/1260 and single point for all others on Columns 1 and 2) ICV: 12/18/12 @ 04:57 (680-260421/17) per Run Log (page 166). ICV results were not included in the data package. CCV: 12/21/12 @ 15:52 (1016/1260) CCV: 12/22/12 @ 00:30 (1016/1260) 	
19. Were calibration results within laboratory/project specifications? 8082A <ul style="list-style-type: none"> ICAL (Criteria: Minimum 5-point curve for PCB 1016 and 1260, single point for all other PCB; ≤20 mean %RSD) <ul style="list-style-type: none"> If %RSD>20, then J-flag positive results and UJ-flag non-detects CCV (Criteria: ≤20%D) 	✓				

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
○ If %D>20, then J-flag positive results and UJ-flag non-detects					
20. Sample results were confirmed using a second gas chromatograph column of dissimilar stationary phase and the %D between results was less than 40 for all detected analytes.	✓			Second column confirmation conducted; however, target analytes were not detected in the sample.	
21. Was a LCS prepared for each batch and matrix?	✓				
22. Were LCS recoveries within lab control limits? If no, J-flag positive results when %R >Upper Control Limit (UCL) and J/R-flag results when %R <Lower Control Limit (LCL).	✓				
23. Were LCS/LCSD RPD within lab specifications? If no, J-flag positive results and UJ-flag non-detects	✓				
24. Was a MS/MSD pair extracted at the proper frequency (one per 20 samples per batch)?	✓				
25. Is the MS/MSD parent sample a project-specific sample?		✓		680-85771-3 (Batch), MS/MSD	
26. Were MS/MSD recoveries within laboratory/project specifications? <i>Only QC results for project samples are evaluated.</i> <ul style="list-style-type: none"> If the native sample concentration > 4x spiking level, then an evaluation of interference is not possible. If either MS or MSD recovery meets control limits, qualification of data is not warranted. MS and MSD %R<10: J and R Flag positive and ND results, respectively MS and MSD %R >10 and <LCL: J-Flag positive and UJ-flag non-detect results MS and MSD R% >UCL (or 140): J-Flag positive results 			✓		
27. Were laboratory criteria met for precision during the MS/MSD analysis? <i>Only QC results for project samples are evaluated.</i> <ul style="list-style-type: none"> If the native sample concentration > 4x spiking level, then an evaluation of interference is not possible. If %RPD > UCL, J-flag positive result and UJ-flag non-detect result 			✓		
28. Were surrogate recoveries within lab/project specifications? <ul style="list-style-type: none"> If %R for 1 Acid or BN surrogates <10, then J-flag positive and R-flag non-detect associated sample results If 2 or more Acid or BN %R >UCL, then J-flag positive 		✓		8082A: The recovery of decachlorobiphenyl (50 and 51%) was below laboratory control limits (54-133%) during the analysis of sample CV0705C-GS (special sample) (680-85785-24) on columns 1 and 2. Non-	UJ

Data Validation Checklist (Continued)

Review Questions	Yes	No	N/A	Samples (Analytes) Affected/Comments	Flag
results <ul style="list-style-type: none"> If 2 or more Acid or BN %R $\geq 10\%$, but $<LCL$, then J-flag positive results and UJ-flag non-detect results If 2 or more Acid or BN, with 1 %R $>UCL$ and 1 %R $\geq 10\%$, but $<LCL$, then J-flag positive results and UJ-flag non-detect results 				detect sample results are estimated (UJ).	
29. Were internal standard (IS) results within lab/project specifications? <ul style="list-style-type: none"> If IS area counts are less than 50% of the midpoint calibration standard, then J-flag positive and UJ-flag non-detect associated sample results If IS area counts are greater than 100% of the midpoint calibration standard, then J-flag positive results If extremely low area counts are reported or performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated, J-flag positive and R-flag non-detect results If retention time of sample's internal standard is not within 30 seconds of the associated calibration standard, R-flag associated data. The chromatographic profile for that sample must be examined to determine if any false positives or negatives exists. For shifts of large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Positive results need not be qualified as R, if mass spectral criteria are met. 	✓				
30. Were lab comments included in report?	✓			Refer to Attachment A (Case Narrative)	
Comments: The data validation was conducted in accordance with the <i>Non-Industrial Use Property Sampling Event QAPP for the 35th Avenue Removal Site, Birmingham, Alabama, Revision 1</i> (OTIE, October 2012). The data review process was modeled after the <i>USEPA Contract Laboratory Program (CLP) National Functional Guidelines (NFG) for Organic Methods Data Review</i> (EPA, October 1999) and <i>USEPA CLP NFG for Low Concentration Organic Methods Data Review</i> (EPA, June 2001). Sample results have been qualified based on the results of the data review process (Attachment B). Criteria for acceptability of data were based upon available site information, analytical method requirements, guidance documents, and professional judgment.					

DV Flag Definitions:

- J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- R The sample results are unusable. The analyte may or may not be present in the sample.
- U The analyte was analyzed for, but was not detected above the associated level; blank contamination may exist.
- UJ The analyte was not detected above the limit, and the limit is approximate and may be inaccurate or imprecise.

ATTACHMENT A
CASE NARRATIVE

CASE NARRATIVE

Client: Oneida Total Integrated Enterprises LLC

Project: 35th Avenue Superfund Site

Report Number: 680-85785-5

With the exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. In addition all laboratory quality control samples were within established control limits, with any exceptions noted below. Each sample was analyzed to achieve the lowest possible reporting limit within the constraints of the method. In some cases, due to interference or analytes present at high concentrations, samples were diluted. For diluted samples, the reporting limits are adjusted relative to the dilution required.

Calculations are performed before rounding to avoid round-off errors in calculated results.

All holding times were met and proper preservation noted for the methods performed on these samples, unless otherwise detailed in the individual sections below.

RECEIPT

The samples were received on 12/14/2012; the samples arrived in good condition, properly preserved and on ice. The temperatures of the 2 coolers at receipt time were 5.2° C and 5.6° C.

PESTICIDES AND PCBS

Sample CV0705C-GS (680-85785-24) was analyzed for Pesticides and PCBs in accordance with EPA SW-846 Method 8081B_8082A. The samples were prepared on 12/18/2012 and analyzed on 12/21/2012.

This method incorporates 2nd column confirmation. Corrective action is not taken for surrogate/spike compounds unless results from both columns are unacceptable. Results outside criteria are qualified.

DCB Decachlorobiphenyl recovered outside the surrogate recovery criteria for CV0705C-GS (680-85785-24).

No difficulties were encountered during the Pesticides and PCBs analysis.

All quality control parameters were within the acceptance limits.

METALS (ICP)

Samples HP0019C-CS (680-85785-8), HP0241A-CS-SP (680-85785-10), CV0507A-CS-SP (680-85785-15), CV0511AB-GS (680-85785-29), CV0511S-CS (680-85785-52), HP0019C-CS (sieve) (680-85785-65), HP0241A-CS-SP (sieve) (680-85785-66), CV0507A-CS-SP (sieve) (680-85785-67), CV0511AB-GS (sieve) (680-85785-68) and CV0511S-CS (sieve) (680-85785-69) were analyzed for Metals (ICP) in accordance with EPA SW-846 Method 6010C. The samples were prepared on 12/17/2012 and 12/18/2012 and analyzed on 12/21/2012.

Several analytes recovered outside the recovery criteria for the MS/MSD of sample HP0019C-CSMSD (680-85785-8) in batch 680-260674, CV0511AB-GS (680-85785-29) in batch 680-260674, and CV0511S-CSMSD (680-85785-52) in batch 680-260674 .

Refer to the QC report for details.

No other difficulties were encountered during the metals analyses.

All other quality control parameters were within the acceptance limits.

TOTAL MERCURY

Samples HP0019C-CS (680-85785-8), HP0241A-CS-SP (680-85785-10), CV0507A-CS-SP (680-85785-15), CV0511AB-GS (680-85785-29), CV0511S-CS (680-85785-52), HP0019C-CS (sieve) (680-85785-65), HP0241A-CS-SP (sieve) (680-85785-66), CV0507A-CS-SP (sieve) (680-85785-67), CV0511AB-GS (sieve) (680-85785-68) and CV0511S-CS (sieve) (680-85785-69) were analyzed for total mercury in accordance with EPA SW-846 Method 7471A. The samples were prepared and analyzed on 12/17/2012.

No difficulties were encountered during the mercury analyses.

All quality control parameters were within the acceptance limits.

HEXAVALENT CHROMIUM

Samples HP0019C-CS (680-85785-8) and HP0019C-CS (sieve) (680-85785-65) were analyzed for hexavalent chromium in accordance with EPA SW-846 Method 3060A/7196A. The samples were prepared on 12/19/2012 and analyzed on 12/20/2012.

Chromium, hexavalent recovered outside the recovery criteria low for the MS of sample HP0019C-CSMS (680-85785-8) in batch 680-260599.

Refer to the QC report for details.

Sample HP0019C-CS (680-85785-8)[10X] required dilution prior to analysis. The reporting limits have been adjusted accordingly.

ATTACHMENT B
QUALIFIED SAMPLE RESULTS

FORM I
GC SEMI VOA ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Savannah Job No.: 680-85785-5
 SDG No.: 68085785-5
 Client Sample ID: CV0705C-GS Lab Sample ID: 680-85785-24
 Matrix: Solid Lab File ID: z120086.d
 Analysis Method: 8081B/8082A Date Collected: 12/12/2012 14:56
 Extraction Method: 3546 Date Extracted: 12/18/2012 21:09
 Sample wt/vol: 15.23(g) Date Analyzed: 12/21/2012 22:07
 Con. Extract Vol.: 5(mL) Dilution Factor: 1
 Injection Volume: 2(uL) GC Column: CLP I ID: 0.32 (mm)
 % Moisture: 23.0 GPC Cleanup: (Y/N) N
 Analysis Batch No.: 260827 Units: ug/Kg

CAS NO.	COMPOUND NAME	RESULT	Q	RL	MDL
12674-11-2	PCB-1016	42	UJ	42	3.7
11104-28-2	PCB-1221	86	UJ	86	6.1
11141-16-5	PCB-1232	42	UJ	42	4.2
53469-21-9	PCB-1242	42	UJ	42	3.6
12672-29-6	PCB-1248	42	UJ	42	9.2
11097-69-1	PCB-1254	42	UJ	42	2.9
11096-82-5	PCB-1260	42	UJ	42	8.6

CAS NO.	SURROGATE	%REC	Q	LIMITS
2051-24-3	DCB Decachlorobiphenyl	50	X	54-133
877-09-8	Tetrachloro-m-xylene	63		46-130

Sample results have been qualified by URS in accordance with the Non-Industrial Use Property Sampling Event QAPP for the 35th Avenue Removal Site, Birmingham, Alabama, Revision 1 (OTIE, October 2012)